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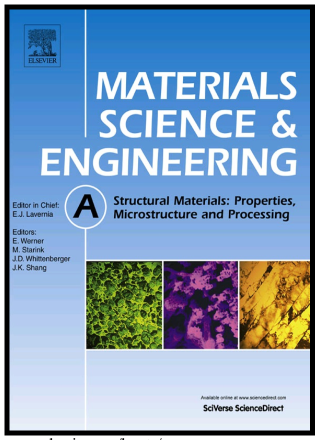

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Fabrication and in situ compression testing of Mg micropillars with a nontrivial cross section: Influence of micropillar geometry on mechanical properties

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Abstract

Micropillars with a nontrivial cross-sectional shape but constant cross-sectional area were fabricated from a pure magnesium single crystal with (0001) orientation by a focused gallium ion beam using a modified annular milling method. The basic mechanical properties (compressive modulus, strength at different plastic strain levels and hardening exponent) of those structures were determined under compression by means of in situ nanoindentation in scanning electron microscope and correlated by the micropillar cross-sectional circumference. It was observed that the modulus and strength increased with increasing circumference. The values of the modulus for the complex cross sectional shapes are on average higher by 5 %, and the yield strength, ranging between 274 MPa and 342 MPa, is on average higher by 20 % relative to micropillars with a simple circular or polygonal cross section. Surprisingly, the hardening exponent remains nearly constant regardless of the micropillar cross section. Finally, an alternative milling method was introduced that gave similar results to the one used, but generally supports much more robust and versatile control over the milling process.

Keywords: electron microscopy; nanoindentation; magnesium alloys; plasticity.

1. Introduction

Modern techniques in materials science allow routine access to materials on the micrometer and submicrometer scales. The diverse structures at such dimensions are of the utmost interest due to their physico-chemical properties [Nal04, Kim12]. Particularly, the measurement of nano- and micromechanical properties is often achieved by means of nanoindentation [ISO15]. Nanoindentation is a versatile approach that comprises indentation and scratch tests of a material's near-surface layers (usually performed with a cono-spherical or wedge indenter tip) and the compression, tension and bending of micrometric and even sub-micrometric objects (often performed with a flat-ended or spherical indenter tip). Such objects typically take the form of micropillars, cantilevers and beams and are most often created by a focused ion beam (FIB) technique [Sel79, Sud88]. The FIB, usually equipped with a gallium (Ga) liquid metal ion source (LMIS), allows fabrication of advanced 3D microstructures and shapes by precise, site specific removal of the material with a nanometric precision independent of the material composition [Gia05].

Micropillars for compression testing are routinely fabricated by either annular (also called top-down) milling [Uch03] or variations of lathe milling [Uch05, Kuz14]. The starting point for both approaches is a micropillar blank in the center of an annular trench of predefined dimensions, milled into the bulk material with a Ga^+ ion beam oriented perpendicular to the sample surface. During annular milling, the Ga^+ ion beam remains perpendicular to the sample surface, and thin layers of material (annular cross section) are gradually removed from the outer wall of the blank. During each step, the beam current and annulus diameters are reduced, resulting in the desired micropillar dimensions. The lower the beam current, the lower the beam diameter, which results in a generally better defined micropillar shape. The lathe milling method uses a Ga^+ beam of a given current tilted by a nonzero angle with respect to the blank longitudinal axis, typically more than 50°, to remove a single side segment from the blank at a time, followed by a sample rotation. The desired micropillar is therefore fabricated on top of the blank. After the given number of milling and rotation steps, the resulting polygonal micropillar resembles that made by annular milling.

Simple annular milling provides a well-defined micropillar cross-sectional shape (CSS) that may be much more complicated than that produced by lathe milling. However, as a result of the beam profile and competing, geometrically constrained sputtering and redeposition, the micropillar base and height are not very well defined, and also, the micropillars are typically tapered by 2°-5° [Uch09, Kie09]. Since the incident angle of the beam is nearly zero, the Ga^+ ions penetrate only the top-most layer that is 1-10 nm thick ([Uch09] and unpublished simulations in the SRIM code [SRIM]). The roughness of the micropillar walls seems to be less than or similar to the thickness of the Ga implanted layer [Kie07, Bai13]. Minimal damage, shallow Ga implantation and mechanical properties of pure Ga lead to the presumption that the implanted Ga negligibly influences the mechanical properties of micropillars fabricated by annular milling [Gre06].

Conversely, an indisputable advantage of lathe milling is the well-defined micropillar base, height and ideal lack of taper when a large tilt angle and small angular steps are used. However, lathe milling is limited to the fabrication of polygonal CSSs only. When the milling process is not adjusted properly, even lathe milling can produce tapered micropillars with both a positive and negative taper. Control over the micropillar CSS regularity may also become less effective due to frequent sample